PAKISTAN STANDARD SPECIFICATION

FOR

BLACK TEA (1st Revision)

0. FOREWORD

This Pakistan Standard was adopted by the Pakistan Standards Institution on 05.10.2000 on the endorsement by the Agriculture & Food Products Divisional Council of the draft finalized by the Tea Sectional Committee.

Pakistan Standards Institution had laiddown the National Standard specification PS: 493 For Black Tea , which covers Black Tea. The Committee felt it necessary that separate standard for Black Tea should be revised.

This standard applies to processed leaves, buds and tender stalk of the dirtinct varieties of Camellia sinensis belonging to the genus camellia prepared by the usual trade practice, but excludes tea waste.

For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated expressing the result of a test or analysis shall be rounded off in accordance with PS:103-1991 (1st Rev) Methods of rounding off Numerical values, the number of significant places retained in the rounded off value shall be the same as that of the specified value in the standard.

This standard is intended chiefly to cover the technical provisions relating to tea and it does not include all the necessary previsions of a contract.

6. SCOPE

This Pakistan Standard prescribes the requirements and methods of test for tea intended for use as a drink.

7. TERMINOLOGY

Black Tea

Black tea shall be fermented, dry and sound, and shall contain no foreign matters, tea waste or other impurities. It may contain stalk out not unfermented stalk exceeding 10 percent by weight which shall be determined by the method prescribed in Appendix-J

Tea Waste

Tea waste is the fine fluff of light or dark colour which produced from plucked ten leaf in course of manufacture.

4. SAMPLING

Samples shall be drawn by any competent authority when considered necessary by the Pakistan Standards Institution.

In sampling, the sampling instruments and the container for samples shall be clean, dry and free from adventitious contamination.

The sample container shall be sealed air-tight after filling and marked with full detail of sampling date of sampling any batch or code number and any other important particulars of the consignment.

Samples shall be drawn from the cube root of containers of each batch of the material. Sample from each selected container shall be not less than 0.5 kg.

Test samples in three shall be prepared from such samples (see clasue 4.4) by transferring 1.0 kg to 1.5 kg of the material which, if necessary, should be ground to pass through No.30 sieve and kept in thoroughly dried tin or glass container and sealed air-tight. The test sample shall be labeled with all the particulars of sampling as given in clause 4.3

One set of test sample shall be sent to the purchaser and one to the vendor and the third set bearing the seals of the purchaser and the vendor or any other authority as detailed by the Pakistan Standards Institution shall constitute the refer sample to be used in case of dispute. It shall be kept at place agreed to between the purchaser and the vendor or as desired by any authority detailed by the Pakistan Standards Institution for this purpose.

5. REQUIREMENTS

The cup quality of tea shall be assessed by the Sample adjudication Panel of the Ten Tenders Association of Pakistan. For this purpose the tea shall be prepared by the method given in appendix-I.

When sampled in the manner prescribed in clause 4 and tested according to the methods prescribed in Appendices mentioned in column 4 of Table -1 the material shall conform to the requirements prescribed in the Table.

S.#	Characteristic	Requirement Black Tea	Method of Test Ref. to Appendix
i	Water Extract, percent by wt min.	33	A
ii	Total Ash, percent by weight	3-8	B
Iii	Ratio of soluble ash to total Ash, percent by weight, Min	45	C
iv	Alkalinity of soluble Ash, calculated as K_2O, percent by wt.	1.4 – 2.0	D
v	Ash insoluble in acid percent by weight, Max	0.8	E
vi	Crude Fiber, percent by wt. Max	15	F
vii	Caffeine, percent by weight, Min	2.5	G
viii	Tennin, percent by weight, Max	10	H
ix	Heavy Metal (Lead) (Pb) max	1mg / kg	-

6. TESTS

Tests of black Tea shall be carried out according to the methods prescribed in Appendix-I, and also in the Appendices mentioned in column 4 of Table-1. In addition , the tea shall
be tested according to the method prescribed in Appendix –J to determine the amount of un-fermented stalk.

7. PACKING AND MARKING

Tea from the garden shall be packed in chests and sacks.

Every such container shall be securely closed and suitably marked to give the following information:-

a. Name and address of the Manufacturer/Packer.
b. Name of the Manufacturer;
c. Batch or code number;
d. Net weight in kg.

APPENDIX – A

(Table –I Item (i)

A-O Determination of Water Extract

A-1 To 2 g of the oven dry sample in a 500 ml volumetric flask add 200 ml of hot water and boil over a low flame for one hour rotating occasionally. Close the flask with a rubber stopper through which passes a glass tube, 75 cm long, for condenser. Boil very slowly so that no stem escapes from the top of the air condenser. Cool, dilute the volume mix thoroughly, and filter through dry filter paper. Transer a 50 ml aliquot to a wigihed dish and evaporate to dryness on steam bath. Place in an oven, heat for one hour at 100 ^{o}C , cool weigh and express the result as the percentage of water extract.

APPENDEX – B

(Table –I Item (ii)

B-0 Determination of Total Ash;

B-1 Heat 10 g of the oven-dry sample in a 50-100 ml platinum or other suitable dish at 100 ^{o}C until water is expelled; add a few drops of pure olive oil, heat slowly over flame until swelling ceases. Place the dish in a muffle furnace at about 525 ^{o}C and leave until white ash is obtained. Moisten ash with water dry on a steam bath and then on hot plate and reash in the muffle furnace at 525 ^{o}C to constant weight and express the result as the percentage of total ash (preserve the total ash for subsequent use)

APPENDEX C

(Table -1, Item (iii)

C-0 Determination of the Ratio of soluble ash to total Ash

C-1 Add water to the ash (see B-1), in the platinum or other suitable dish, heat nearly to boiling, filter through ashless filter paper, and thoroughly was with hot water until combined filtrate and washing measure about 60 ml. Return the paper and the contents

to the dish and ash in the muffle furnace, calculate the ratio of water-soluble ash (percent by weight) by subtracting the percentage of water insoluble ash from the percentage of total ash (see B-1) (preserve the filtrate and insoluble ash for subsequent use)

APPENDIX - D

(Table -1 Item (iv)

D-0 Determination of Alkalinity of soluble Ash

D-1 Reagents

D-1.1 Methyl orange indicator.

D-1.1.1 Dissolve 0.5 g Methyl orange in water and dilute to one litre.

D-1.2 Hydrochloric acid, 0.1 N (see PS: 37 Hydrochloric acid).

D-2 Procedure

D-2.1 Cool filtrate from C-1 and titrate with 0.1 HCl using methyl orange indicator. Express the alkalinity in terms of normal acid per 100 g of the sample. The alkalinity of soluble ash is also calculated as K_2O using the following formula.

1 ml of 0.1 N acid – 0.0047 g of K_2O

APPENDIX – E

(Table -1, Item-V)

E-0 Determination of Ash Insoluble in Acid.

E-1 Procedure

E-1.1 Boil total ash (See B-1) with 25 ml of HCl (1+2.5) for 5 minutes covering the dish with watch glass to prevent spattering collect the insoluble matter on a gooch or ashless filter, wash with hot water until washings are acid-free, ignite until carbon-free, cool, weigh and express the result as the percentage of ash insoluble in acid.

APPENDIX – F

(Table -1, Item Vi)

F-0 Determination of crude fibre

F-1 Apparatus

F-1.1 Condenser use double surface condenser that will maintain constant volume of solution through out digestion.

F-1.2 Digestion flask – Use digestion flasks of such size and shape that the solution will not be less than 2.5 cm not more than 3.75 cm in depth (700-750) ml Erlermeyer flask is recommended).

F-1.3 Filtering cloth : Use filtering cloth of such quality that permits no solid matter to pass through

F-1.4 Gooch Crucible

F-1.5 Fluted glass funnel

F-1.6 Desiccator

F-1.7 Air Oven

F-1.8 Electrical Muffle furnace

F-2 Reagents

F-2.1 Sulphuric acid solution – 0.225 N (1.25 g of H_2SO_4 in 100 ml of water).

F-2.2 Sodium Hydroxide solution – 0.312 N (1.25 g of sodium hydroxide A. R grade, in 100 ml of water).

F-2.3 Asbestos – Gooch grade medium fibre, acid alkali and water washed and then ignited.

F-3 Procedure

F-3.1 Extract 2 g of the oven dry material with a ether and transfer the residue, together with 0.5 g of asbestos to a digestion flask.

Add 200 ml boiling H_2SO_4 Solution, immediately connect the digestion flask with a condenser and heat. It is essential that the contents of the flask come to boiling within one minute and the boiling continues briskly for exactly 30 minutes.

Rotate the flask frequently until the sample is thoroughly wetted. Take care to keep the material form remaining on the sides of the flask out of contact with the Solution. (A blast of air conducted into the flask will serve to reduce the frothing of the liquid) at the expiration of 30 minutes, remove the flask immediately, filter through lines in a fluted funnel and with boiling water until washing are no longer acidic. Bring a quantity of NaoH solution to boiling and keep at this temperature under reflux condenser until used. Wash charge and asbestos back into the flask with 200 ml of boiling NaOH solution using wash bottle marked to deliver 200 ml (boiling NaOH solution is conveniently transferred to the wash bottle by means of bent tube through which the liquid is forced by blowing into the tube connected with the top of the reflux condenser attached to NaOH flask).

Connect the flask with the reflux condenser and boil exactly for 30 minutes.

F-3.2 After expiration of 30 minutes, remove the flask and immediately fiter through a gooch crucible prepared with asbestos mat through Alundum crucible or through the filtering cloth in fluted funnel. If filtering cloth is used, thoroughly wash the residue with boiling water and transfer to a gooch crucible prepared with a thin but close layer of ignited asbessos. After thorough washing with boiling water, was with 15 ml of alcohol. Dry the

crucible and contents at 110°C to constant weight. Cool in a desiccator and weigh. Incinerate the contents of the crucible in the electric muffle furnace or over Maker burner at dull red heat until the carbonaceous matter has been consumed (20 minutes). Cool in a desiccators and weigh. Report loss in weight as cruide fibre. Calculate the percentage.

APPENDIX – G

(Table –I, Item vii)

G-0 Determination of Caffeine:

G-1 Procedure:

G-1.2 Into a weighed one litre Erlenmeyer weigh 5 g of the oven dry sample. Add about 500 ml of water, Swirl and heat to boiling. Add 10 g of heavy mg boil gently over a lower flame for 2 hours with occasional shaking. Add water to prevent frothing and to wash down the sides of the flask. Cool and make to weight with water (tare weigh + 510 g + weight of the sample). Filter, collect 200 ml of clear filtrate (equivalent to 0.4 of the weight of the sample, and 20 ml of H_2SO_4 (1+9), and transfer to a 500 ml separator. Shake 6 times with Chloroform, using 25, 20, 15, 10 and 10 ml portions. Treat the combined extracts with 5 ml of 1 percent Potassium hydroxide solution, when the liquids separate completely drain the chloroform layer into a Kjeldahl flask. Wash he alkaline solution into the separater with two 10 ml portions of chloroform and combine the washings with the remaining bulk of the extract. Evaporate or distill the chloroform to less then 25 ml.

Transfer the sample to a digestion flask. Add 0.7 g of mercuric oxide of 0.65 g of metallic mercury, 15 g of powdered potassium Sulphate or anhydrous Sodium Sulphate, and 25 ml of concentrated H_2 SO_4 (to amount of H_2SO_4 may be increased, if necessary). Place the flask in an inclined position and heat gently until frothing ceases (if necessary add a small amount of pureffin to reduce frothing); boil briskly until the solution clear and then for at least 30 minutes long.

Cool, add about 200 ml of water, cool below 25 °C add 25 ml sodium of potassium Sulphite or 80 g of Sodium thisulphate in 1 litre of water and mix to precipitate the mercury. Add a few zinc granules to prevent bumping, tilt the flask, and add a layer of sodium hydroxide (25 g of solid reagent or enough solution to make the contents a strongly alkaline) without agitation. (Thiosuphate or Sulphide solution may be mixed with the Sodium hydroxide solution before addition to the condenser, and with the tip of the condenser immersed in a standard acid in the receiver, rotate the flask to mix contents thoroughly then heat until all Ammonia has distilled (at least 150 ml of distillate). Titrate the excess standard acid in the distillate with a standard alkali solution, using methyl red. Correct for flank determination on reagents.

1ml of 0.1 N H_2SO_4 = 4.85 mg of anhydroux caffeine.

APPENDIX – H

(Table –I, Item – viii)

H-0 Determination of Tannin

H-1 Reagents;

H-1.1 Potassium permanganate solution-prepare a solution containing 1.33 g per litre and obtain its equivalent in term of 0.1 N Oxalic acid.

H-1.2 Indigo carmine solution – prepare a solution containing 6 g of indigo carmine (free form Indigo blue) and 50 ml of H_2SO_4 per litre.

H-1.3 Gelatin solkution – Soak 25 g of gelatin for 1 hour in the standard Nacl solution heat until gelatin dissolves, cool and dilute with standard NaCl solution to 1 litre.

H-1.4 Acid sodium chloride solution – Acidify 975 ml of standard NaCl solution with 25 ml of H_2SO_4.

H-2 Procedure

H-2.1 Boil 5 g of the oven dry sample for 30 minutes with 40 ml waer, coo, transfer to a 500 ml volumetric flask and dilute to the mark. To 10 ml of the infustion (filtered, if not clear) add 25 ml of the indigo carmine solution and abouit 750 ml of water. Add the $KMnO_4$ solution from the burette, little at a time, with stirring, until the colour changes to light green than dropwise until colour changes to bright yellow or to fain pink at rim. Designate ml of $KMnO_4$ used as .

Mix 100 ml of the clear infusion of tea with 50 ml of the gelatin solution, 100 ml of the acid NaCl solution, and 10 g of powdered kaolin, and shake for several minutes in a stoppered flask. Allow the mixture to settle and decent through filter. Mix 25 ml of filterate with 25 ml of the indigo carmine solution and about 750 ml water, and titrate with $KMnC_4$ before Ml of $KMno_4$ used subtracted from that obtained about “a” gives the quantity of $KMno_4$ required to oxidize the tannin. 1 ml of 0.1 oxalic acid g = 0.0042 g tannin (gallotannic acid).

APPENDIX – I

(Clause 5.1)

I-0 Determination of cup Quality

I-1 Procedure

I-1.1 Pour 100 ml of boiled water on to 3 g of the oven dry sample and allow it to infuse for 5 minutes, then pour of the liquor.

Note the odour of the infused tea and report mustiness if any.

APPENDIX – J

(Clause 3.1.1)

J-0 Determination of unfermented materials

J-1 Procedure:

J-1.1 Spread 5 g of dry sample on a plastic or glass sheet and pick out the unfermented materials, if any, by forceps. Weigh the unfermented materials and express the result in percentage on oven dry basis.